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(54) **Liquid bleaching laundry detergent composition**

(57) A liquid substantially anhydrous heavy duty laundry detergent composition comprising a suspension of builder salt, sodium perborate monohydrate and solid activator therefor, all ground together to reduce the particle size of its solids to less than about 10 microns.

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SPECIFICATION

Liquid bleaching laundry detergent composition

5 The present invention relates to nonaqueous liquid heavy duty laundry detergents. 5

In accordance with one aspect of the present invention, a highly effective and stable nonaqueous liquid heavy duty laundry detergent is prepared by dispersing a solid builder salt, sodium perborate monohydrate and a solid activator for the perborate in a substantially anhydrous liquid nonionic surfactant and grinding the dispersion to reduce the particle size of those solids to less than about 10 microns, e.g. to an average particle size of 2 to 10 microns or even lower, such as 1 micron. Preferably, in the ground product, less than about 20% by weight (more preferably less than about 10% by weight) of the suspended particles have diameters above 10 microns. 10

It will be understood that as the particle size is decreased, by grinding, fresh reactive surfaces are exposed and the surface area available for reaction is increased. Also, the energy involved in the fine grinding operation necessarily causes localized rises in temperature within the mixture being ground (even though the grinding mill is cooled, e.g. by cold water running through a jacket of the mill, during grinding). Despite this, the compositions of the present invention are highly stable, showing little, if any, loss of bleaching activity during grinding or on storage. 15

In the grinding operation it is preferred that the proportion of solid ingredients be high enough (e.g. at least about 40% such as about 50%) so that the solid particles are in contact with each other and are not substantially shielded from one another by the nonionic surfactant liquid. Mills which employ grinding balls (ball mills) or similar mobile grinding elements have given very good results. Thus, one may use a laboratory batch attritor having 8 mm diameter steatite grinding balls. For larger scale work a continuously operating mill in which there are 1 mm or 1.5 mm diameter grinding balls working in a very small gap between a stator and a rotor operating at a relatively high speed (e.g. a CoBall mill) may be employed; when using such a mill it is desirable to pass the blend of nonionic surfactant and solids first through a mill which does not effect such fine grinding (e.g. a colloid mill) to reduce the particle size to less than 100 microns (e.g. to about 40 microns), prior to the step of grinding to an average particle diameter below about 10 microns in the continuous ball mill. 20 25 30

It is preferred that the solid builder salt be an alkali metal polyphosphate such as sodium tripolyphosphate ("TPP"). Other suitable polyphosphates include the pyrophosphates such as tetrasodium pyrophosphate and metaphosphates. In place of all or part of the polyphosphate one or more other detergent builder salts may be used, such as alkali metal carbonates or bicarbonates, silicates, or organic builders such as polycarboxylates. Typical suitable builders are those disclosed in U.S. Patents 4,316,812; 4,264,466 and 3,630,929. 35

The preferred activator is tetraacetylenediamine ("TAED"). Another suitable activator is glucose pentaacetate. Other suitable activators are those disclosed in U.S. Patent 4,264,466 or in column 1 of U.S. Patent 4,430,244. Polyacylated compounds are preferred activators. 40

The invention may be put into practice in various ways and a number of specific embodiments will be described to illustrate the invention with reference to the accompanying examples. All proportions in this specification and claims are by weight unless otherwise indicated.

45 **EXAMPLES 1A and 1B** 45

Example 1B is a comparison example.

Table 1 below gives the ingredients and proportions for Examples 1A and 1B. These compositions are each ground for 30 minutes at room temperature in a Wieneroto W-1.S attritor with 8 mm diameter steatite balls, producing a liquid suspension in which the average particle size of the ground material is below about 10 microns in each case. 50

TABLE 1

Examples	1A	1B	
Ingredients	Proportions		
5 Nonionic surfactant	35	32.57	5
Acid-converted nonionic surfactant	16	16	
TPP	30	30	
10 Sodium salt of ethylenediamine tetramethylene phosphonic acid (EDITEMPA)	1.5	1.5	10
15			15
Examples	1A	1B	
Ingredients	Proportions		
20 Copolymer of methacrylic acid and maleic anhydride completely converted to sodium salt form			20
25 (Sokalan CP5)	4	4	25
Sodium carbonate	2.5	2.5	
Sodium perborate monohydrate			
30 (NaBO ₃ H ₂ O)	4.5	--	30
Sodium perborate tetrahydrate	--	6.93	
TAED	5	5	
35 Optical brighteners	0.5	0.5	35
Enzyme slurry (Protease slurried in nonionic surfactant)	1	1	
40			40
Each composition contains 0.72% active O ₂ and the content of unbound water of the ingredients is less than 1%. After 48 hour aging at 35°C, the composition of Example 1A is still liquid while the composition of Example 1B has become substantially solid (its yield stress has increased to a value greater than 20 Pascals). After 5 days aging at 35°C, analysis for available			
45 peracetic acid indicates that the effective loss of TAED on grinding and aging is 7% for the composition of Example 1A and 45% for the composition of Example 1B.			45
The nonionic surfactant used in these examples comprises a mixture of equal parts of:			
(a) a relatively water soluble nonionic surfactant which forms a gel when mixed with water at 25°C, specifically a C ₁₃ to C ₁₅ alkanol which has been alkoxyated to introduce 10 ethylene oxide and 5 propylene oxide units per alkanol unit and			
50			50
(b) a less water-soluble nonionic surfactant, specifically a C ₁₃ to C ₁₅ alkanol which has been alkoxyated to introduce 4 ethylene oxide and 7 propylene oxide units per alkanol unit.			
The acid-converted nonionic surfactant is a half ester of succinic acid and a nonionic surfactant (specifically ethoxylated C ₁₂ to C ₁₅ alkanol having about 5 ethylene oxide units per molecule of alkanol). This half ester can be produced by the reaction of succinic anhydride and			
55			55
the nonionic surfactant at elevated temperature (e.g. 60°C) preferably in the presence of a small amount of catalyst such as pyridine. The resulting acid-terminated product acts in the compositions as an antigelling agent. It reduces the temperature at which the nonionic surfactant forms a gel with cold water and thus aids in the dispensing of the compositions			
60			60
during operation of automatic home laundry machines at winter temperatures. Other carboxylic acid materials soluble in the nonionic surfactant may be used for this purpose.			
Th TPP is a blend of anhydrous TPP and a small amount of TPP hexahydrate such that the chemically bound water content is about 3%, which corresponds to about one H ₂ O per pentasodium triphosphosphate molecule. Such TPP may be produced by treating anhydrous TPP			
65			65
with a limited amount of water. The presence of the hexahydrate slows down the rapid rate of			

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solution of the TPP in the wash bath and inhibits caking. One suitable TPP is sold under the name Thermphos NW; the particle size of this TPP as supplied is in the neighbourhood of 400 microns; its phase I content is about 60%.

- In preparing the mixture before grinding it is preferred to add the solid ingredients to the nonionic surfactant, with the TPP being added last.
- The mixture dispenses readily with cold water in an automatic washing machine. Its specific gravity is about 1.25. It gives excellent washing when used at a dosage of about 100 grams per wash load (as compared with 170 grams per wash load for the usual heavy duty laundry detergent powders) in conventional European Home laundry machines (which employ about 20 litres of water for the washing bath) in which the water is heated, during washing to about 60°C.

EXAMPLE 2

A ground composition is prepared as in Example 1 from the ingredients in the stated proportions given in Table 2:

Ingredients	Proportions
nonionic surfactant	40.8
TPP	49.8
Ethylene diamine tetraacetic acid	1.2
TAED	4.5
Sodium perborate monohydrate	3.6
Hydroxylamine sulphate	0.1

- The hydroxylamine sulphate acts to inhibit the undesirable bleach-decreasing action of the enzyme, catalase, present in soiled clothes.
- As is well known, the nonionic synthetic surfactants are characterised by the presence of an organic hydrophobic group and an organic hydrophilic group and are typically produced by the condensation of an organic aliphatic or alkyl aromatic hydrophobic compound with ethylene oxide (hydrophilic in nature). Practically any hydrophobic compound having a carboxy, hydroxy, amido or amino group with a free hydrogen attached to the nitrogen can be condensed with ethylene oxide or with the polyhydration product thereof, polyethylene glycol, to form a nonionic detergent. The length of the hydrophilic or polyoxyethylene chain can be readily adjusted to achieve the desired balance between the hydrophobic and hydrophilic groups. Typical suitable liquid nonionic surfactants are those disclosed in U.S. Patents 4,316,812 and 3,630,929, as well as those described and listed in the discussion of nonionic surfactants in Kirk-Othmer "Encyclopedia of Chemical Technology", 3rd Edition, Vol. 22 (1983) pages 360 to 379.
- The activator usually interacts with the peroxygen compound to form a peroxyacid bleaching agent in the wash water. It is preferred to include a sequestering agent of high complexing power to inhibit any undesired reaction between such peroxyacid and hydrogen peroxide in the wash solution in the presence of metal ions. Such a sequestering agent is an organic compound which is able to form a complex with Cu^{2+} ions, such that the stability constant (pK) of the complexation is equal to or greater than 6, at 25°C, in water of an ionic strength of 0.1 mole/litre, pK being conventionally defined by the formula: $\text{pK} = -\log K$ where K represents the equilibrium constant. Thus, for example, the pK values for complexation of copper ion with NTA and EDTA at the stated conditions are 12.7 and 18.8, respectively. Suitable sequestering agents include the sodium salts of nitrilotriacetic acid (NTA); ethylene diamine tetraacetic acid (EDTA); diethylene triamine pentaacetic acid (DTPA); diethylene triamine pentamethylene phosphonic acid (DTPMP); and ethylene diamine tetramethylene phosphonic acid (EDITEMPA).
- Other ingredients which may be included in the composition are enzymes (e.g. proteases, amylases or lipases or mixtures thereof); optical brighteners, antiredeposition agents (e.g. sodium carboxymethylcellulose), and colourants (e.g. pigments or dyes).
- The composition may also contain an inorganic insoluble thickening agent or dispersant of very high surface area such as finely divided silica of extremely fine particle size (e.g. of 5-100 millimicrons diameter such as sold under the name Aerosil) or the other highly voluminous inorganic carrier materials disclosed in U.S. Patent 3,630,929, in proportions of 0.1-10%, e.g. 1 to 5%. It is preferable, however, that the compositions be substantially free of such compounds and of other silicates, it has been found, for instance, that silica and silicate promote an undesired decomposition of the peroxyacid formed in the wash bath.
- In the compositions of the present invention, typical proportions of the ingredients are as

follows:

Suspended detergent builder, within the range of about 10 to 60%, such as about 20 to 50%, e.g. about 25 to 40%;

5 Liquid phase comprising nonionic surfactant (and, optionally, dissolved carboxylic acid material), within the range of about 30 to 70%, such as about 40 to 60%; this phase may also include a diluent such as a glycol, e.g. polyethylene glycol (e.g. "PEG 400") or hexylene glycol. 5

Carboxylic acidic antigelling agent, in the range of about 0.01 to 1 part per part of nonionic surfactant, such as about 0.05 to 0.6 part, e.g. about 0.2 to 0.5 part;

10 Sodium perborate monohydrate, in the range of about 2 to 15%, such as about 4 to 10%; Activator in the range of about 1 to 8%, such as about 3 to 6%. 10

The unbound moisture content of the composition should be less than about 2% preferably below about 1%, such as about 0.5% or less.

15 While it is most convenient and effective to grind all the solid ingredients together it is within the broader scope of the present invention to grind separate suspensions of individual ingredients or groups of ingredients and combine these together after such grinding. 15

In this application all proportions are by weight unless otherwise indicated. In the Examples, atmospheric pressure is used unless otherwise indicated.

20 It is understood that the foregoing detailed description is given merely by way of illustration and that variations may be made therein without departing from the spirit of the invention. 20

CLAIMS

1. A substantially anhydrous liquid heavy duty laundry detergent composition comprising particles of solid sodium perborate monohydrate, particles of solid activator for the perborate, and particles of solid builder salt, all dispersed in a liquid nonionic surfactant, the sizes of the said particles being less than about 10 microns. 25

2. A composition as claimed in Claim 1 in which the activator comprises tetraacetylene-diamine.

3. A composition as claimed in Claim 1 or Claim 2 in which the builder salt comprises pentasodium tripolyphosphate. 30

4. A composition as claimed in Claim 1, 2 or 3 produced by grinding a suspension in the said liquid of the said solids, the said solids having particle sizes of at least 40 microns before the said grinding.

5. A composition as claimed in any one of Claims 1 to 4 comprising about 10 to 60% of the said builder salt, about 2 to 15% of sodium perborate monohydrate and about 1 to 8% of activator. 35

6. A composition as claimed in Claim 5 in which the amount of the said builder salt is about 25 to 40%.

7. A composition as claimed in Claim 6 in which the activator comprises tetraacetylene-diamine, the said composition having been produced by grinding a suspension of the said solids in the said liquid, the said solids having particle sizes of at least 40 microns before the said grinding. 40

8. A composition as claimed in Claim 7 substantially free of silica or silicates.

9. A composition as claimed in Claim 1 substantially as specifically described herein with reference to Example 1A or 2.